Influence of Several Nano Minerals on The Mechanical Properties of Fresh and hardened Concrete

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Abstract— **The current study aims at obtaining the influence of adding several nano-minerals on the mechanical properties of fresh and hardened concrete. Four types of Nano minerals were investigated in this work; namely: Brown Iron Oxide, Barium Sulfate, Titanium Dioxide and Poly Acrylic Acid. Five contents of each nano-minerals were studied. A range of contents starting from 0.5 % to 2.5 % with an increment of 0.5 % by cement content of Brown Iron Oxide and Barium Sulfate was investigated. The Titanium Dioxide and Poly Acrylic Acid were added to concrete in percentages of 1 % to 5 % with an increment of 1 % by cement content. Adding 5 % Poly Acrylic Acid to the other three nano-minerals at selected contents separately was also studied. The measured mechanical properties of concrete comprised slump value, 7 and 28 days compressive strength. Some of the results revealed that there was 24 % increase in the slump value corresponding to every 1 % increase in PAA content. An improvement of 6.9 % in the 28 days compressive strength was measured at every 0.5 % increase of Brown Iron Oxide. It was also found that by adding 5 % Poly Acrylic Acid and 2.5 % Brown Iron Oxide to the concrete at the same time, the slump increased from 90 mm to 170 mm (88.9 % increase) and the 28 days compressive strength improved from 37.37 kg/cm^2 to 49.35 kg/cm^2 (32.1 % increase).**

Keywords-Nano-Minerals, Brown Iron Oxide, Barium Sulfate, Titanium Dioxide and Poly Acrylic Acid, Concrete Strength, Slump.

I. BACKGROUND

The recent studies on nano-materials and nano-technologies have indicated the potential use of these materials in various fields of civil engineering, especially in concrete technology. This is due to the fact that concrete is a macro-material strongly influenced by its nano-properties. The use of nano materials would improve the various characteristics of concrete, such as workability, strength, durability [1,2,3,4].

However, there is still a lack of knowledge concerning the suitable nano-materials for construction and their behavior; a lack of specific standards for design and execution of the construction elements using nano-materials; a lack of detailed information regarding the nano-products content; high costs; the unknows of health risks associated with nano-materials. Therefore more researches are to be conducted to overcome this lack of knowledge and information [1,4].

Nano-material is defined as materials with at least one external dimension in the size range from approximately 1-100 nanometers. Nanoparticles are objects with all three external dimensions at the nano scale. Engineered nano-particles are intentionally produced and designed with very specific properties related to shape, size, surface properties and chemistry. The behavior of nanomaterial may depend more on surface area than particle composition itself. The relative-surface area is one of the principal factors that enhance its reactivity, strength and electrical properties [5,6].

Shekari and Razzaghi, [7] concluded that nano particles can be very effective in improvement of both mechanical properties and durability of concrete. The use of nano ZrO_2, Fe_3O_4, TiO_2 and Al_2O_3 with constant content enhanced the mechanical properties of fresh and hardened concrete as compressive strength, indirect tensile strength and durability. Jalal, Fathi and Farzad [8] indicated that the use of 15 % fly ash and 5% TiO_2 by cement weight enhances the mechanical properties of fresh and harden concrete as concrete flow, durability, compressive strength, flexure strength, and indirect tensile strength.

Karatasios, Kilikoglou, Theoulakis, Colston and Watt [9] showed that the use of Barium hydroxide in concrete increases the durability of hardened concrete against sulphate attack. Mermerdaş, Güneyisi, Gesoğlu, Özturan [10] proved that adding meta-kaolin in concrete mixture decreases the drying shrinkage and weight loss of concrete. Hyun-Soo, Jae-Yong and Myoung-Youl [11] added 4 % Iron Oxide by cement content to the concrete mixture. An improvement in the compressive and flexural strength of concrete was measured. Al Mishhadani, Ibrahem and Naji [12] studied the effect of nano-metakaolin on compressive strength, splitting tensile strength of concrete. The ordinary Portland cement was substituted by 3%, 5% and 10% by cement weight of nano-metakaolin. The results indicated that the compressive strength and splitting tensile strength of concrete with nano-metakaolin were significantly higher than that of the corresponding control values.

Yu, Spiesz and Brouwers [13] investigated the effect of nano-silica on the hydration and microstructure development of Ultra-High Performance Concrete (UHPC) with a low binder amount. They found with the addition of nano-silica, the hydration of cement can be promoted and more C–S–H gel can be generated. They also noted that the nano-silica allows more air entrapped in the mix and hence increase in porosity of the hardened concrete. They concluded that there should be an optimum amount of nano-silica for the production of UHPC with the lowest porosity, at which the positive effect of the nucleation and the negative influence of the entrapped air can be balanced.

Sharobim and Mohammedin [14] investigated the effect of Nano-liquid on the mechanical and physical properties of hardened concrete such as water permeability, absorption, abrasion resistance, compressive strength, indirect tensile strength and flexural strength. Nano-liquid was sprayed on the dry surface of hardened concrete specimens two days before testing. The results show that Nano-liquid can reduce the water absorption and coefficient of permeability of concrete. Also, it can improve the abrasion resistance of concrete, but it has no effect on concrete strength.

II. AIM AND OBJECTIVES

The aim of the current study is to obtain the influence of several Nano minerals on the mechanical properties of fresh and hardened normal strength concrete. Four types of Nano mineral materials were investigated in this work; namely: Brown Iron Oxide ($Fe_2O_3/Fe_3O_4 \geq 93\%$), Barium Sulfate ($BaSO_4$) and Poly Acrylic Acid ($(C_8H_8.C_3H_4O_2)_x$). The tested mechanical properties of concrete comprised slump value, 7 days and 28 days compressive strength.

III. EXPERIMENTAL PROGRAM

An experimental program was design to achieve the aim and objective of the current research. Normal strength concrete was utilized to investigate the effect of the selected Nano minerals (NMs) on concrete. The following paragraphs explain the properties of the selected NMs and the concrete constituents.

A. Nano Mineral

Four types of NM materials were selected for the current work, namely: Brown Iron Oxide ($Fe_2O_3/Fe_3O_4 \geq 92$ %), Barium Sulfate ($BaSO_4$), Poly Acrylic Acid ($[C_8H_8.C_3H_4O_2]_x$) and Titanium Dioxide (TiO_2). Table I presents the characteristics of these four NM materials.

Table I. Characteristics of the Selected NM materials

Item	Brown Iron Oxide	Barium Sulfate	Titanium Dioxide	Poly Acrylic Acid
Appearance	Brown	white	white	White
Toxicity	Not toxic	Not toxic	Not toxic	Not toxic
Residue on 45 µm	0.30	-	0.05 max	-
Normal Size, nm	< 100	< 100	<100	<100
PH-Value	7	6.5-8.0	7 - 8.5	9-10
Density, g/ cm^3	4.8	4.5	4.23 to 3.78	1.08

Iron oxide is a Nano mineral which has particles diameters ranging between 1 and 100 nanometers. The two main forms of iron are magnetite (Fe_3O_4) and its oxidized form magnetite (γ-Fe_2O_3). Iron oxides produced from ferrous sulfate by heat soaking, removal of water, decomposition, washing, filtration, drying and grinding. They produced in either anhydrous or hydrated forms. Their range of hues includes yellows, reds, browns and blacks. The one which used in this study is the Brown Iron Oxide (BIO)

The second NM minerals which applied in this work is the Barium Sulfate, $BaSO_4$ (BS). It is s white crystalline solid which is odorless and insoluble in water. It occurs as the mineral barite, which is the main commercial source of barium and materials prepared from it. The white opaque appearance and its high density was exploited in its main applications.

The Poly Acrylic Acid (PAA) is component from pure acrylic emulation, which used in paint manufacturing. In this research, the product XL-800 is used which has good weather resistance, pulverization resistance and color change resistant properties. This is because the polymer membrane has excellent balance between internal cohesion and adhesion.

The Titanium Dioxide (TiO), also known as titanium (IV) oxide or titania, is the naturally occurring oxide of titanium. Generally it is sourced from Ilmenite, Rutile and Anatase.

B. Concrete Consitutients

The physical and chemical characteristics of the Portland cement were performed. The results of the cement tests were conformed with the standards of ASTM C191 [15] , C109 [16] and C204 [17]. The initial setting time and final setting time were 105 minutes and 315 minutes, respectively. The Blain fineness was 3035 cm^2/g. The 3 and 7 days mortar compressive strength were 13.5 MPa and 29.6 MPa, respectively.

The sieve analysis of fine and coarse aggregate was made according to ASTM C136 [17], the result was listed in the Table II. The fineness modulus of sand is 2.504. The average Saturated Surface Dry (SSD) specific gravity is 2.61. The 24 hours absorption of coarse and fine aggregates were 1.51% and 1.27 %, respectively. The Flakiness, soundness and elongation of coarse aggregate were found to satisfy the relevant ASTM standards. Drinkable water was used in preparation of all performed concrete mixes.

Table II. Size Analysis of Fine and Coarse Aggregate

Sieve Size, mm	Crushed Aggregate			
	Coarse			Fine
	Fooliya	Adasiya	Simsimya	Sand
37.5	100.00			
25	92.60			
19	77.44	100.00		
12.5	33.78	56.03	100.00	
9.5	10.27	21.83	95.51	
4.75	0.80	2.33	20.54	
2.36	0.27	0.37	0.71	100.00
1.19				88.40
0.6				41.60
0.15				2.00
0.075				

C. Mix design

The mix proportioning of the normal concrete was carried out based on the ACI 211.1 [18]. The target cubic compressive strength was 30 MPA, the max. aggregate size was 25 mm, the target slump was between 75 to 100 mm. The coarse and fine aggregate were blended to satisfy the ASTM gradation curves. Table III includes the adjusted proportions of the normal concrete mixes.

D. Testing Program

The influence of the selected NMs on the mechanical properties of fresh and hardened concrete was obtained by adding 5 percentages of each NM materials by cement content in addition to the control mix (0% NM). Table IV presents the proportions of the NM added to the control mix.

Table III. adjusted Proportions of Concrete Mixes

Item	Weight, Kg/ 1m^3	Volume, m^3
Entrapped air	0	0.0150
Water/cement	0.569	-
Water	182	0.1650
Cement	320	0. 1016
Coarse aggregate	1161	0.4519
Fine aggregate	687	0.2665
Total	**2350**	1.000

Two additional mixes were prepared by adding 5% of PAA material by cement weight to the control mix. Then, 2.5 % of BIO by cement weight was added to the first mix and 2.5 % of BS by cement weight to the second mix. This was carried out in order to investigate the effect of using more than one NM at a time.

Table IV. Proportions of NM with the Control Mix

	Mix	BIO	BS	PAA	TiO
Constituents		5 mixes for each NM			
% NM, 0.5% or 1 % increment (inc.)	0%	0.5 to 2.5 (0.5 % inc.)		1 to 5 (1 % inc.)	
NM weight, kg	-	1.6 to 8.0		3.2 to 16	
Cement, kg	320	320		320	
Water, kg	182	182		182	
Fooliya, kg	468	468		468	
Adasiya, kg	374	374		374	
Simsimiya, kg	318	318		318	
Sand, kg	687	687		687	

E. Mixing and Curing Process

The concrete was mixed according to Standard Method of Making and Curing Test Specimens in the Laboratory ASTM C 192 [19]. The dry NMs were blended first with the cement powder, while the liquid NM was added to the mixing water. The 100×100×100 mm cubes were used to prepare the concrete specimens. All concrete specimens were left in their molds for about 20 to 40 hours. Then, they stripped off from the molds and put in the standard curing tank at 20 ^{o}C ± 4^{o}C until time of testing.

F. Testing of Specimens

The fresh properties of the prepared concrete mixes, with and without NM were obtained by measuring the slump value in accordance with ASTM C143[20]. The 7, 14 and 28 days compressive strengths were carried out on standard cubes of size 100 mm using 3000 kN compressive testing machine in accordance with ASTM C39 [21]. The compression test was performed using a loading rate ranging between 0.15 to 0.35 MPa/s.

IV. Results and Discussion

The slump, 7 and 28 days compressive strength were measured and discussed, as presented in the following paragraphs.

A. Slump Values

The slump values of the concrete specimens having BIO, BS, PAA and TiO NM are presented in Fig. 1. The slump of concrete specimens with 1 % BIO and 1 % BS by cement weight do not show any significant increase, beyond which a 6 % increase in slump was measured for every percent increase in BIO and BS. A similar finding was observed with the addition of TiO.

A significant increase in slump was measured in concrete specimens with PAA. An average of 24% increase in slump with every percent increase in PAA in concrete was obtained. Therefore, the optimum percent of PAA with concrete with depend on the degree of workability required by the job.

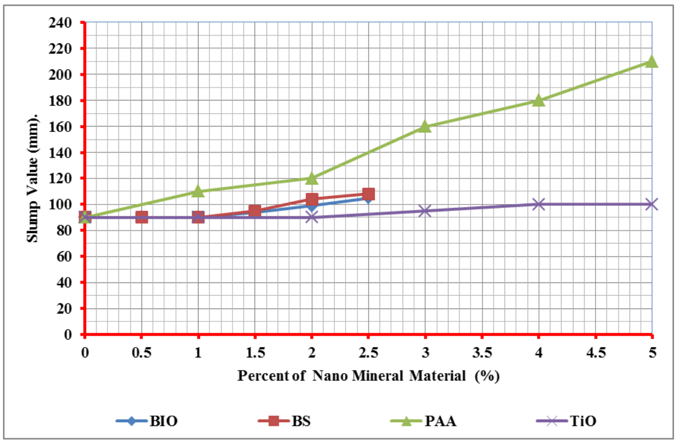

Fig. 1. Slump of BIO, BS, PAA and TiO Mixs

B. 7 Days Compressive Strength

The 7 days compressive strengths of the concrete specimens having BIO, BS, TiO and PAA are shown in Fig. 2. Fig. 2 indicated that there was an increase of about 6.4 % and 3.4 % in the 7 days compressive strength of concrete specimens at every 0.5 % increase in BIO and BS respectively. This finding was observed at the five increments of BIO and BS, namely, 0.5, 1, 1.5, 2 and 2.5 % contents.

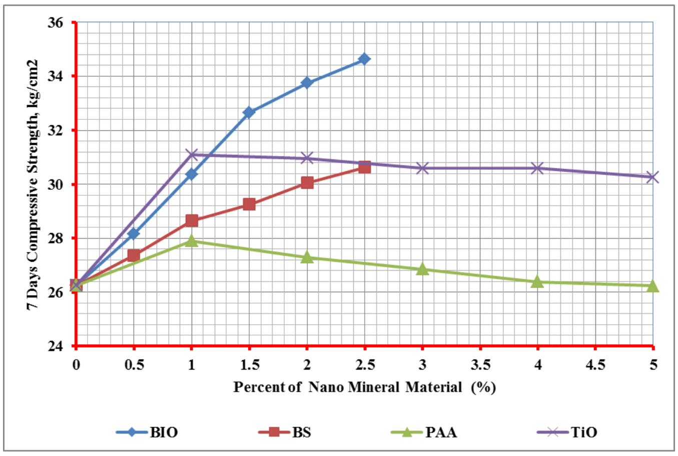

Fig. 2. The 7 Days Compressive strength of NM Mixes.

An addition of 1 % TiO and 1 % PAA by cement weight to concrete gave 18.5 % and 6.3 % improvement in the 7 days compressive strength, respectively. However, this gain in strength started to decrease beyond 1 % content of TiO and 1 % PAA with a decreasing rate of 1.34 % and 0.7 % respectively at each 1% increment. At the maximum percent used of TiO and PAA (5 % by cement weight), the strength was equal to or slightly above the control value. It means that there was no overall decrease in the strength.

C. 28 Days Compressive Strength

Fig. 3 presents the 28 days compressive strength of the BIO, BS, TiO and PAA concrete specimens. An improvement of 6.9 % and 3.0 % in the 28 days strength was obtained at every percent increase of BIO and BS in the concrete, respectively. In the current study, The total improvement obtained in the 28 days strength at 2.5 % of BIO and BS by cement weight was 34.3 % and 15.2 %, respectively.

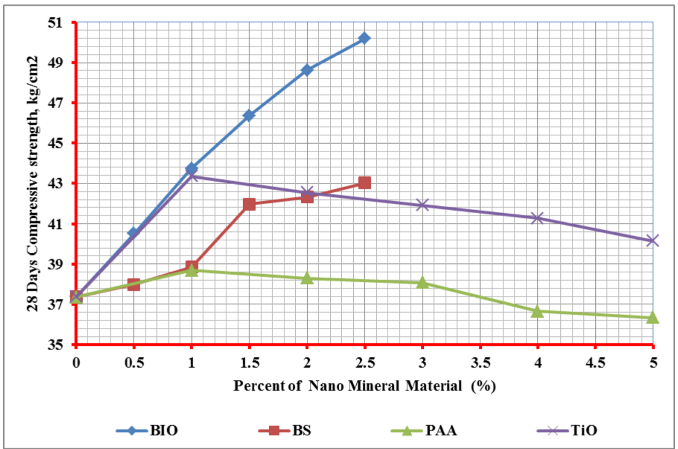

Fig. 3. The 28 Days Compressive strength of NM Mixes.

Similar to the results of the 7 days strength, the maximum strength was obtained at 1% PAA and 1 % TiO by cement weight in the concrete, i.e. 3.6 % and 16 %, respectively. As PAA and TiO contents increased by 1 % increments up to 5 %, there was a loss in the gained strength. At 5 % content of TiO, the 28 days strength was still higher than that of the control specimen (without NM) by 7.5 %. At about 3.5 % of PAA, the strength which was gained at 1% content was lost, and the strength was equal to the strength of the control specimen.

D. Results of Adding PAA to Other N-M materials

The effect of adding two of the applied NM at a time on the concrete properties was obtained. These samples comprised specimens with 5 % PAA + 2.5 % BIO, specimens, 5 % PAA + 1 % TiO and specimens with 5 % PAA + 2.5 % BS. The idea behind using 5 % PAA in these specimens is to benefit from the significant influence of PAA on slump. The slump, 7 and 28 days compressive strength of the specimens having two NMs at a time are indicated in Table VI.

Table VI reveals that when adding 5 % PAA to 2.5 % BIO, the slump enhanced from 105 mm (for BIO alone) to 170 mm, without losing the obtained improvement in the 28 days compressive strength. It means that by adding 5 % PAA to the 2.5 % BIO speciemns, the slump increased from 90 mm to 170 mm (88.9 % increase) and the 28 days compressive strength improved from 37.37 kg/cm^2 to 49.35 kg/cm^2 (32.1 % increase).

Table VI. Slump and 28 Days Compressive Strength of BIO+PAA, TiO+PAA and BS+PAA Specimens

Concrete mix	Slump, mm	28 days compressive strength, kg/cm^2
Control mix	90	37.37
	BIO + PAA	
2.5 % BIO	105	50.19
5% PAA	210	36.34
2.5 % BIO + 5% PAA	170	49.35
	TiO + PAA	
1 % TiO	100	40.15
5% PAA	210	36.34
1 % TiO + 5% PAA	145	44.12
	BS + PAA	
2.5 % BS	108	43.03
5% PAA	210	36.34
2.5 % BS + 5% PAA	150	39.29

Table VI showed also the results obtained when adding 5 % PAA to the 1 % TiO specimen (which is the optimum content for this type of NM). The slump was increased from 100 mm (for TiO alone) to 145 mm (45 % enhancement). At the same time, the 28 days compressive strength increased from 40.15 kg/cm^2 to 44.12 kg/cm^2 (9.9 % increase. In total, adding 5 % PAA to the 1 % TiO specimen increased the slump from 90 mm to 145 mm (61.1 % increase) and the 28 days compressive strength improved from 37.37 kg/cm^2 to 44.12 kg/cm^2 (18.1 % increase). Finally, The results of adding 5 % PAA to the 2.5 % BS specimen are also presented in Table VI. In this case, the slump increased from 108 mm (for BS alone) to 150 mm with 5 % PAA and 2.5 % BS. A slight reduction of 8.6 % in the 28 days compressive strength was observed when adding 5 % PAA to the 2.5 % BS specimen. However, adding 5 % PAA to the 2.5 % BS specimen slightly enhanced the 28 days from 36.34 kg/cm^2 to 39.29 kg/cm^2 (8.1 % increase).

V. CONCLUSIONS

The influence of several nano-minerals on the fresh and hardened properties of concrete was obtained. These nano-minerals comprised Brown Iron Oxide, Barium Sulfate, Titanium Dioxide and Poly Acrylic Acid. Five contents of each nano-mineral were studied. A range of contents starting from 0.5 % to 2.5 % by cement weight with an increment of 0.5 % of Brown Iron Oxide and Barium Sulfate was investigated separately. The Titanium Dioxide and Poly Acrylic Acid were also added separately to concrete in percentages from 1 % to 5 % by cement weight with an increment of 1 %. The addition of 5 % Poly Acrylic Acid to the other three nano-minerals separately at selected contents was also studied. The following conclusions can be drawn from the analysis of the measured results:

1- The slump of the concrete specimens with 1 % BIO and specimens with 1 % BS by cement weight do not show any significant change, beyond which an increase in slump of about 6 % was measured for every percent increase in BIO and BS.

2- A similar finding of those specimens with BIO and specimens with BS was also observed with the addition of TiO.
3- A significant increase in slump was measured in concrete specimens with PAA. An average of 24% increase in slump with every percent increase in PAA was obtained.
4- There was an also increase of 6.4 % and 3.4 % in the 7 days compressive strength of concrete at every 0.5 % increase in specimens with BIO and specimens with BS respectively.
5- The addition of 1 % TiO and 1 % PAA by cement weight to concrete gave 18.5 % and 6.3 % improvement in the 7 days compressive strength, respectively. This gain in strength started to decrease beyond the 1 % TiO and 1 % PAA with a decreasing rate of 1.34 % and 0.7 % respectively at every 1% increment of these NM.
6- An improvement of 6.9 % and 3.0 % in the 28 days strength was measured at every percent increase of BIO and BS to the concrete, respectively. The total improvement obtained in the 28 days strength at 2.5 % of BIO and 2.5 % BS by cement weight was 34.3 % and 15.2 %, respectively.
7- The maximum strength of 3.6 % and 16 % for the PAA specimens and TiO specimens was obtained at 1% content of each of them, respectively. Beyond this 1 % content of PAA and TiO, there was a loss in the gained strength.
8- The addition of 5 % PAA to the 2.5 BIO specimens increased the slump from 90 mm to 170 mm (88.9 % increase) and improved the 28 days compressive strength from 37.37 kg/cm^2 to 49.35 kg/cm^2 (32.1 % increase).
9- By adding 5 % PAA to the 1 % TiO specimens, the slump increased from 90 mm to 145 mm (61.1 % increase) and the 28 days compressive strength improved from 37.37 kg/cm^2 to 44.12 kg/cm^2 (18.1 % increase).
10- Adding 5 % PAA to the 2.5 % BS specimens gave an increase in slump from 108 mm (for BS alone) to 150 mm with 5 % PAA and 2.5 % BS together. Also, a slight enhancement of 8.1 % was obtained in the 28 days compressive strength.

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